

# **Anisotropy in Hydrogenated Amorphous Silicon Films as Observed Using Polarized FTIR-ATR Spectroscopy**

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# ANISOTROPY IN HYDROGENATED AMORPHOUS SILICON FILMS AS OBSERVED USING POLARIZED FTIR-ATR SPECTROSCOPY

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## ABSTRACT

We used polarized attenuated total reflection (ATR) measurements together with Fourier transform infrared (FTIR) spectroscopy to investigate the vibrational spectra of hydrogenated amorphous silicon (a-SiH<sub>x</sub>) films 0.5-1.0 microns in thickness. We deposited the films using hot-wire or plasma-enhanced chemical vapor deposition methods (HWCVD or PECVD, respectively) on crystalline silicon and cadmium telluride substrates. Our ATR technique gave a spectral range from 2100-400 cm<sup>-1</sup>, although the Si-H wagging mode absorption band at 640 cm<sup>-1</sup> was somewhat distorted in the a-SiH<sub>x</sub>/Si samples by impurity and lattice absorption in the silicon ATR substrates. We report the identification of a SiO<sub>2</sub> impurity band with maximum intensity at 1240-1230 cm<sup>-1</sup> in p-polarization. The assignment of this band to SiO<sub>2</sub> vibrations is supported by secondary-ion mass spectrometry (SIMS) measurements. Our polarized FTIR-ATR spectra of HWCVD and PECVD a-SiH<sub>x</sub> films on <111> Si ATR substrates show that the impurity dipoles are oriented strongly parallel to the film growth direction. The wagging mode absorbance band is more intense in the film plane. This trend is less pronounced for the Si-H stretching vibrations. These observations are consistent with some degree of anisotropy or medium-range order in the films. The anisotropy in the Si-H bands may be related to residual stress in the films. Our scanning electron microscopy (SEM) analyses of the samples offer additional evidence of bulk structural anisotropy in the a-SiH<sub>x</sub>/Si films. However, the SiO<sub>2</sub> impurity band was not observed in the polarized ATR-FTIR spectra of the a-SiH<sub>x</sub>/CdTe samples, thus indicating that the Si substrates influence formation of the impurity in the a-SiH<sub>x</sub>/Si films.

## INTRODUCTION

Infrared (IR) spectroscopy and hydrogen secondary-ion mass spectrometry (SIMS) are the two most accessible methods for determining the hydrogen content of hydrogenated amorphous silicon (a-SiH<sub>x</sub>) films [1,2]. For IR transmission spectroscopy, the a-SiH<sub>x</sub> films are commonly deposited on crystalline silicon substrates. However, in IR transmission, the electric field vector of the transmitted radiation is aligned predominantly with the sample plane.

Attenuated total reflection (ATR) infrared spectroscopy involves the use of an IR-transparent waveguide of refractive index  $n_1$ , in contact with an IR-absorbing sample of refractive index  $n_2$ , where typically  $n_1 > n_2$ . If infrared light is directed into the waveguide (substrate) at an incidence angle ( $\theta$ ) greater than its critical angle, total internal reflection takes place, except at wavelengths where the evanescent standing wave extending into the sample interacts with the IR-active dipoles and is attenuated [3]. The ATR-IR spectrum of a sample resembles its IR absorbance spectrum, except for the wavelength dependence in the ATR-absorbance band intensities induced by the wavelength-dependent penetration depth of the evanescent wave into the rarer sample medium [3]. However, in cases where  $n_2$  nearly equals or slightly exceeds  $n_1$ , as with a-SiH<sub>x</sub> films on Si or CdTe substrates in air, total internal reflection takes place at the sample outer surface (Fig. 1), rather than at the substrate/sample interface. In these cases there is no wavelength dependence of the ATR-absorbance band intensities [3],

although dispersion in  $n_2$  may have some effect on the ATR-IR absorbance band shapes of  $\alpha$ - $\text{SiH}_x$  films on Si substrates [4]. Unlike transmission spectroscopy, ATR spectroscopy allows polarization of the incident light in the film growth direction, as well as in the film plane (Fig.1).

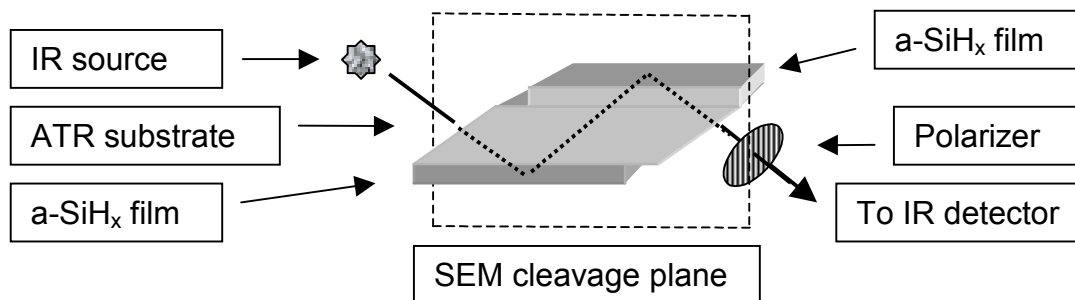


Fig. 1. Schematic drawing of the attenuated total reflection configuration for infrared spectroscopy of  $\alpha$ - $\text{SiH}_x$  films. The cleavage plane for SEM measurements on the  $\langle 111 \rangle$  Si ATR substrates is also shown.

An early work pioneering the application of ATR spectroscopy to the analysis of  $\alpha$ - $\text{SiH}_x$  films [5] described the measurement of the first overtone absorbance of the Si-H stretching vibration near  $3950 \text{ cm}^{-1}$ , as well as the absorbance from  $3900$ - $5400 \text{ cm}^{-1}$  corresponding to high-pressure molecular hydrogen in microvoids in the films. To observe these weak absorbance bands, an optical path of  $6.3 \text{ cm}$  through the Si ATR substrates was used. However, at such long path lengths, impurity, lattice, and free-carrier absorption in the Si substrates limited their transparency to frequencies higher than about  $1500 \text{ cm}^{-1}$  [5,6]. In this work, we used short-path-length ( $0.7$ - $1.4 \text{ cm}$ ) Si and CdTe ATR substrates to extend the range of our spectroscopic measurements to frequencies as low as  $400 \text{ cm}^{-1}$ .

## EXPERIMENT

Thin films of hydrogenated amorphous silicon were deposited on both of the  $\langle 111 \rangle$  faces of crystalline silicon ATR substrates and on one face of polycrystalline cadmium telluride ATR substrates, as shown in Fig. 1. These substrates were blown clean with dry nitrogen before film deposition, but were otherwise used as received from the manufacturer (Harrick Scientific Co.), as is our typical practice in preparing  $\alpha$ - $\text{SiH}_x$  films on Si substrates for IR analysis of H content. The gaps in film coverage of the substrate (Fig. 1) result from the clamp used to secure the substrates to the heater can in the deposition system [1]. The clamp also shielded the  $45^\circ$  faces from the film deposition reaction. The ATR substrates were of the  $45^\circ$  single-pass parallelepiped (SPP) type [4], and were either  $10 \times 5 \times 1 \text{ mm}$  or  $5 \times 5 \times 1 \text{ mm}$  in size. The  $\langle 111 \rangle$  orientation of Si and other cubic semiconductors is commonly used for ATR substrates, since it facilitates producing them in the  $45^\circ$  SPP geometry. We used hot-wire chemical vapor deposition (HWCVD) [1] and plasma-enhanced chemical vapor deposition (PECVD, or glow-discharge) [2] methods to deposit the  $\alpha$ - $\text{SiH}_x$  films. We prepared other HWCVD  $\alpha$ - $\text{SiH}_x$  films on  $\langle 100 \rangle$  crystalline Si wafers. These samples were analyzed both by clamping them to an uncoated  $\langle 111 \rangle$  Si ATR substrate and by IR transmission. A Harrick Model 4XV 4x beam condenser equipped with a Perkin-Elmer wire-grid polarizer was operated with average incidence angle set to  $45 \pm 15$  degrees to the film plane to collect the FTIR-ATR spectra. The wide range of actual incidence angles is caused by the  $\sim F1$  beam condenser optics. The condenser optics minimized the effects of the nominally odd number of IR reflections in the  $5 \times 5 \times 1 \text{ mm}$ ,  $45^\circ$  SPP ATR substrates [7].

The infrared spectra were collected using either a Nicolet Magna 550 FTIR spectrophotometer or a Nicolet 510 FTIR spectrophotometer with deuterated tryglycine sulfate detector. The polarized FTIR-ATR spectra (I) of the films deposited on ATR substrates were taken by averaging  $10^3 - 10^4$  scans of the samples at  $8 \text{ cm}^{-1}$  resolution, and were converted to absorbance units by taking the ratio  $A = -\log_{10}(I/I_0)$ , where  $I_0$  is the unratiod spectrum of the uncoated substrate taken at similar polarization and resolution. The polarized ATR spectra of the films deposited on Si wafer substrates were taken similarly, except that the samples were clamped to an uncoated  $45^\circ$ ,  $10 \times 5 \times 1 \text{ mm}$ , SPP ATR crystal for analysis. Transmittance FTIR spectra ( $100I/I_0$ , or %T) were recorded by collecting 128 scans of the samples (I) and substrates ( $I_0$ ) at  $4 \text{ cm}^{-1}$  resolution and computing the ratio. The transmittance spectra were converted to absorbance units for comparison to the polarized ATR-absorbance spectra or were converted to absorbance coefficient spectra ( $\alpha_\nu$ ) for calculation of hydrogen content [1,2].

After collection of their infrared spectra, some of the samples were subjected to SIMS depth profiling for Si, H, O, C, and/or N. The SIMS depth profiles of the samples were collected using a Cameca IMS-3f system with a  $\text{Cs}^+$  primary-ion source, and detection of negative secondary ions. The diameter of the ion etch pit was  $\sim 100 \mu\text{m}$  at the sample surfaces. Certain of the samples were then cleaved and imaged in cross-section using scanning electron microscopy (SEM). Sample films deposited on the  $\langle 111 \rangle$  faces of Si ATR substrates were cleaved through the  $\langle 111 \rangle$  tilt plane, as indicated in Fig. 1. The SEM images were collected with a JEOL JSM field-emission scanning electron microscope with an effective spatial resolution of  $\sim 1 \text{ nm}$ . Table 1 gives a summary of the analyses done on each sample.

Table 1. Deposition parameters and analytical methods for a-SiH<sub>x</sub> film samples. All films were deposited on both sides of  $5 \times 5 \times 1 \text{ mm}$  Si ATR substrates, unless otherwise noted.

Sample Number	Deposition Method, Substrate Type	T <sub>substrate</sub> (K)	$100*[\text{H}]/[\text{H}+\text{Si}]$ , Si=5E22 $\text{cm}^{-3}$	Thickness ( $\mu\text{m}$ )	Analysis Methods
H473	GD	470.5	10.0** (11.5)	(0.8)	ATR, %T, SIMS, SEM
H497	HW 10x5x1 CdTe, 1-side	541.5	9.3**	1.5 †	ATR, SEM
H498	GD 10x5x1CdTe, 1-side	470.5	10.0** (12.3)	(0.9)	ATR, %T, SIMS, SEM
T896	HW	541.5	9.8** (9.1)	(0.5)	ATR, %T, SIMS
H496	HW, 1-side stepwise deposition	541.5	9.8** (7.6) 14.0*	(0.7)	ATR, %T, SIMS
HW64	HW, 1-side Si wafer	598.0	12.4*	[0.88]	Contact ATR, SEM
HW76	HW, 1-side Si wafer	613.0	8.4*	[0.55]	Contact ATR, SEM
HW58	HW, 1-side Si wafer	648.0	1.7*	[1.50]	Contact ATR, SEM
H350i	HW	669.3	2.2** (2.9)	(0.9)	ATR, %T, SIMS
H472	HW, 1-side bilayer deposition	690.6 (bulk), 541.5 (15 nm)	1.2, 9.3** (0.3 - 0.6, bulk, 15 nm)	(0.9)	ATR, %T, SIMS, SEM
H351I	HW	719.0	0** (0.6)	(4.7)	ATR, SIMS
			*FTIR ( ) SIMS	[ ] profiler ( ) SIMS	
			**estimated from T <sub>sub</sub>	† SEM	

## RESULTS

Fig. 2 shows the polarized ATR-FTIR spectrum of PECVD sample #H473. Our ATR technique enabled detection of the Si-H<sub>x</sub> fundamental stretching band from 2100 - 2000 cm<sup>-1</sup> and a previously unreported SiO<sub>2</sub> impurity band with maximum at 1240 - 1230 cm<sup>-1</sup>, visible only in p-polarization (parallel to the film growth direction). The shape of the wagging mode fundamental band at 640 cm<sup>-1</sup> was distorted by the strong substitutional carbon absorbance at 607 cm<sup>-1</sup> and by the phonon absorbance at 619 cm<sup>-1</sup> in the Si substrates. However, a higher intensity of the 640 cm<sup>-1</sup> band in s-polarization (parallel to the film plane) is evident, indicating some enhancement of the wagging modes in the film plane. The stretching mode band from 2100 - 2000 cm<sup>-1</sup> shows little or no polarization dependence (indicating little or no orientation of the Si-H dipoles), and is about five times more intense than the same band measured in transmission mode because of enhancement by multiple reflections. The polarized ATR-FTIR spectra of the other a-SiH<sub>x</sub>/Si samples qualitatively resemble those shown in Fig. 2.

We considered assigning the impurity band to the first overtone of the fundamental Si-H wagging vibration at 640 cm<sup>-1</sup>, but its intensity is too great, even exceeding that of the supposed parent band in the case of sample # H472, and bears no relationship to the hydrogen content of the samples. However, the intensity of the impurity band does have a strong positive correlation with both the average oxygen (Fig. 3) and the average carbon content of the a-SiH<sub>x</sub> films on Si substrates, integrated from the film surface through the interfacial region to the substrate, as measured by SIMS. We were able to fit the polarization-dependent components of this band with the sum of two Gaussian distributions centered at 1228 and 1165 cm<sup>-1</sup>, respectively (Fig. 4). These frequencies are consistent with Si-O stretching vibrations in a crystalline SiO<sub>2</sub> impurity phase, although carbon as C-O could also be present in the impurity. The strong orientation of the Si-O dipoles in the film growth direction explains why the SiO<sub>2</sub> impurity band has not been observed previously using transmission IR spectroscopy, in which the electric field of the incident radiation is oriented in the film plane. The impurity bands at 1228 and 1165 cm<sup>-1</sup> resemble the bands reported by Chabal, *et al* [8] in the IR spectra of oxidized silicon wafers. There is some indication of the impurity bands in the contact FTIR-ATR spectra (not shown) of samples HW58, HW64, and HW76 (Table 1), but these spectra are noisy because of poor contact between the rigid samples and the equally rigid ATR substrates, and because of an unfavorable optical mismatch [3]. The intensity of a shoulder at 1030 cm<sup>-1</sup> on the band structure in Fig. 4 is invariant with polarization (Fig. 2), and is consistent with an amorphous SiO<sub>x</sub> impurity phase (1 < x < 2). Fig. 5 shows that the intensity of the SiO<sub>2</sub> impurity band is linearly dependent on the deposition time during a stepwise, single-sided HWCVD deposition of sample H496, for which a final film thickness of 0.9 μm was measured.

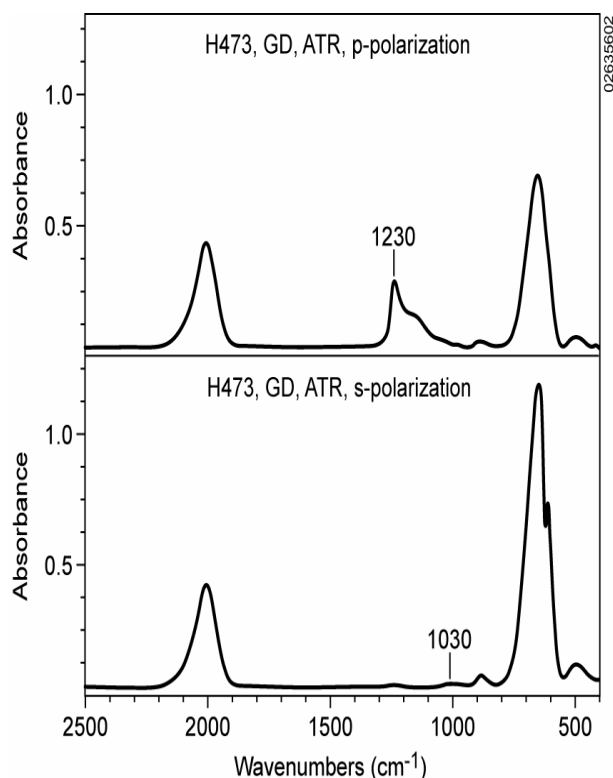


Fig. 2. Polarized FTIR-ATR spectra of a-SiH<sub>x</sub>/Si sample #H473.

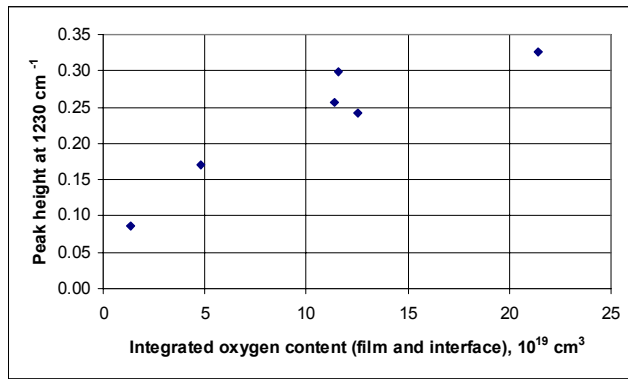


Fig. 3. Relationship between FTIR/ATR band height at  $1230 \text{ cm}^{-1}$  and integrated oxygen content of a-SiH<sub>x</sub>/Si film samples determined from SIMS depth profiles.

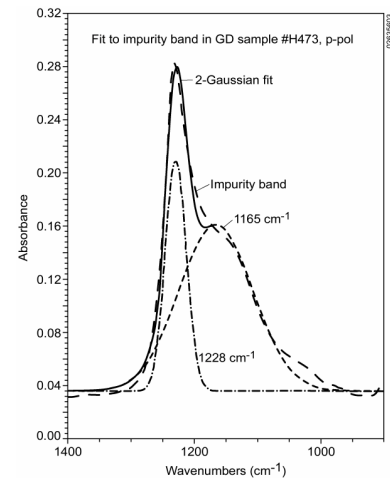


Fig. 4. Two-Gaussian fit to the SiO<sub>2</sub> impurity band observed in the FTIR-ATR spectrum of a-SiH<sub>x</sub>/Si sample #H473, with  $\nu_1 = 1228 \text{ cm}^{-1}$ ,  $\Delta\nu_1 = 39 \text{ cm}^{-1}$ ,  $\nu_2 = 1165 \text{ cm}^{-1}$ , and  $\Delta\nu_2 = 137 \text{ cm}^{-1}$ .

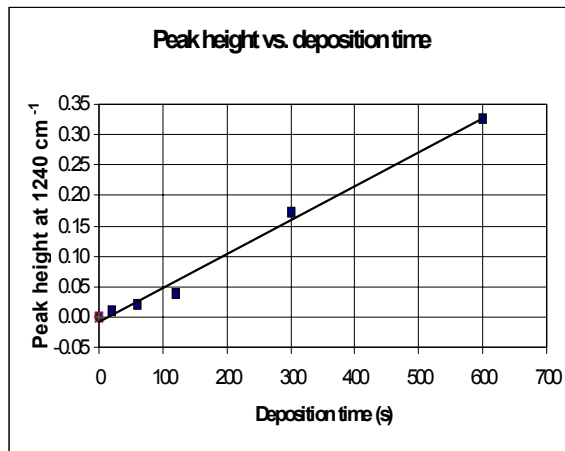


Fig. 5. Intensity of the SiO<sub>2</sub> impurity band at  $1240 \text{ cm}^{-1}$  for p-polarization as a function of deposition time in step-deposited HWCVD sample #H496.

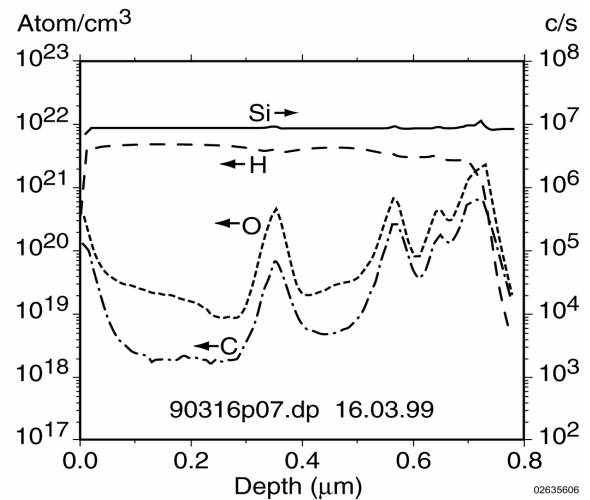


Fig. 6. SIMS Si, H, O, and C depth profile of layer-deposited a-SiH<sub>x</sub>/Si sample #H496.

A SIMS depth profile through sample #H496 (Fig. 6) shows high levels of C and O at the film surface and at the film/substrate interface. These features are common to all the samples analyzed via SIMS. Also evident in Fig. 6 are accumulations of C and O at the individual deposition interfaces. These contaminants apparently accumulated when the sample was removed from the evacuated deposition chamber for FTIR-ATR spectroscopic measurements. It is likely that the O (and possibly C) impurities introduced to the sample surface after each deposition reacted with the silane to yield the oriented SiO<sub>2</sub> impurity phase in each subsequent deposition step during growth of this sample. Consistent with earlier results [9], the SIMS analyses show that there is little penetration of H into the crystalline Si substrates.



An interesting variation in the FTIR data is the complete absence of the  $\text{SiO}_2$  impurity band in the polarized ATR spectra (not shown) of the  $\text{a-SiH}_x$  films deposited on polycrystalline CdTe ATR substrates (H497 and H498, Table 1). Although carbon and oxygen levels in these samples were similar to those observed in some of the  $\text{a-SiH}_x/\text{Si}$  samples, it may also be necessary to have a surface layer rich in  $\text{SiO}_x$  to serve as a precursor to the  $\text{SiO}_2$  impurity. The polarized ATR-FTIR spectra of these samples are otherwise similar to those in Figure 2, except that they show the wagging mode absorbance bandshape at  $640\text{ cm}^{-1}$  without substrate absorption artifacts. The polarized ATR-FTIR spectra of samples H497 and H498 also indicate some enhancement of the wagging absorbance in the film plane. In the SEM image of HWCVD sample #H497 (not shown), which was deposited on CdTe, the  $\text{a-SiH}_x$  film appears amorphous in areas of poor adhesion and structured in areas where it makes good contact with the substrate. The SEM images (not shown) of PECVD  $\text{a-SiH}_x/\text{CdTe}$  sample #H498 reveal no evidence of microstructure, but also show poor film adhesion, perhaps from residual stress.

The SEM micrographs of the cleaved cross-sections of the HWCVD  $\text{a-SiH}_x$  films on silicon substrates show a distinct columnar microstructure and some areas of poor adhesion. The microstructure is especially evident in the SEM image of sample #H472 (Fig. 7), which incorporated a 15-nm  $\text{a-SiH}_x$  interfacial layer nominally 9.3 % in H. However, the SEM micrographs of PECVD  $\text{a-SiH}_x/\text{Si}$  sample #H473 (not shown) indicate that the columnar microstructure is discontinuous in this  $\text{a-SiH}_x$  film.

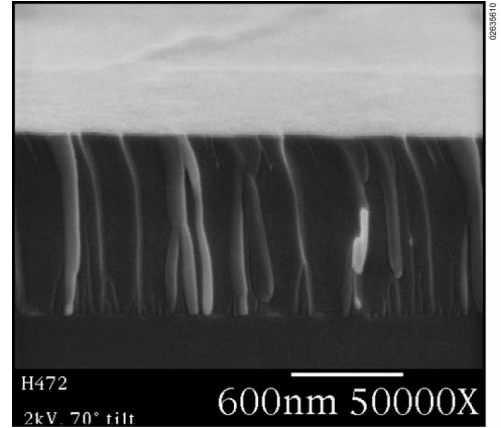


Fig. 7. Cross-sectional SEM micrograph of cleaved  $\text{a-SiH}_x/\text{Si}$  sample #H472.

## CONCLUSIONS

Using polarized FTIR-ATR spectroscopy and SIMS depth profiling, we have demonstrated the existence of an impurity phase, likely to contain  $\text{SiO}_2$  and possibly C-O, and having dipoles highly oriented in the film growth direction in  $\text{a-SiH}_x/\text{Si}$  films. The impurity phase is not observed in  $\text{a-SiH}_x/\text{CdTe}$  films. We have also shown that the Si-H wagging absorbance is enhanced in the film plane in  $\text{a-SiH}_x$  films on  $\langle 111 \rangle$  Si substrates and on polycrystalline CdTe substrates, perhaps because of residual stress or other anisotropic properties of the films. Our SEM measurements support the FTIR-ATR measurements of structural anisotropy in the  $\text{a-SiH}_x/\text{Si}$  films. The anisotropy is influenced more by the substrates, and their surface contaminants, than by the film deposition method. The anisotropy in the wagging absorbance at  $640\text{ cm}^{-1}$  may explain some of the scatter in hydrogen content calibration measurements made using transmission-mode FTIR spectroscopy of  $\text{a-SiH}_x/\text{Si}$  samples [1].

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